EFFECT OF PRETREATMENT PARAMETERS ON COBALT REMOVAL AND SURFACE ROUGHNESS OF TUNGSTEN CARBIDE PRIOR TO DIAMOND COATING

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To my beloved mother and father

Abdullah Bin Ahamad

Saadiah Bt Ismail

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ABSTRACT

Cobalt removal and surface roughness substrate are important parameters to determine the quality of diamond coating. Various methods are adopted to solve this problem and one of them is by using chemical pretreatment. In this present work, an attempt is made to study the effect of pretreatment parameters namely temperature and time on cobalt removal and surface roughness of tungsten carbide. Full factorial experimental design followed by Response Surface Methodology (RSM) is employed in this study to plan and analyze the experiment. The surface roughness Ry and Rz, and cobalt removal are the independent response variables. Empirical models are successfully developed to predict amount of cobalt removal and surface finish of the substrate after single step etching process. Experimental results show that the temperature is the most significant factor followed by the etching time for predicted surface roughness Ry while for surface roughness Rz, the temperature and time^2 are found to be the most significant factors. Whereas for interaction of time and temperature is not significant to influence Ry. It is only significant to determine Rz and cobalt removal.

ABSTRAK

Pembuangan kobalt dan kekasaran permukaan merupakan parameter yang penting untuk menetukan kualiti sesuatu salutan berlian. Pelbagai cara telah diambil untuk menyelesaikan masalah ini dan salah satunya adalah melalui pra-rawatan kimia. Dalam kajian ini, usaha telah dibuat untuk mengkaji kesan parameter rawatan iaitu suhu dan masa ke atas pembuangan kobalt dan kekasaran permukaan ke atas tungsten karbida. Rekabentuk faktor ujukaji penuh diikuti oleh metodologi sambutan permukaan telah digunakan dalam kajian ini untuk merancang dan menganalisis ujikaji. Kekasaran permukaan Ry, Rz dan pembuangan kobalt adalah pembolehubah yang bersandar. Model empirik berjaya dibina untuk meramal jumlah pembuangan kobalt dan kekasaran permukaan substrat selepas langkah tunggal proses pempunaran. Keputusan proses pempunaran menunjukkan bahawa suhu merupakan faktor yang paling signifikan diikuti oleh masa punaran untuk meramalkan kekasaran permukaan Ry manakala untuk Rz, suhu dan masa^2 didapati faktor yang paling signifikan. Sementara perkaitan masa dan suhu adalah tidak signifikan untuk mepengaruhi Ry. Ia hanya signifikan untuk mengira Rz dan pembuangan kobalt.

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LIST OF SYMBOLS

Adeq. precision	-	adequate precision
Adj. R^2	-	adjusted R^2
Cor. Total	-	totals of all information corrected for the mean
CV	-	coefficient of variation d.f. degrees of freedom
Pred. R^2	-	predicted R^2
Prob. > F	-	proportion of time or probability you would expect to get the stated F value
PRESS	-	predicted residual error sum of squares
R^2	-	coefficient of determination
S.D.	-	square root of the residual mean square
SEM	-	Scanning Electron microscopy

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Tungsten carbide is one of the important tools and dies materials because of its high hardness, strength and wears resistance over a wide range of temperatures (Lee and Li, 2001). The amount of cobalt present was significantly affects to properties of tungsten-carbide tools. As cobalt content increases, the strength, hardness, and wear resistance of WC decrease, while its toughness increases because of high toughness of cobalt (Kalpakjian and Schmid 1997). However in diamond coating, the present of the cobalt on the surface was leading to poor of diamond coating quality (Ahmeda *et al*., 2004; Heaney *et al*, 2008; Chattopadhyay *et al*., 2008; Marinkovica, 1999 *et al*.; Mallika, and Komanduri, 1999). The surface roughness was also factor in determining the performance of diamond coating (Polini, 2006).

There are many methods used to reduce the cobalt content and roughening the surface and one of them was chemical pretreatment. Intensive research has been carried out by previous researchers to reduce the cobalt content while roughening the substrate surface on tungsten carbide. Sarangi *et al*. (2008a) conducted a research by comparing between two types of pretreatment namely Murakami's reagent and

$HCl+HNO_3+H_2O$. It was observed the Murakami's reagent roughen the surface substrate while cobalt removal performed by $HCl+HNO_3+H_2O$. Sarangi *et al*. (2008b) also study the effect of $HCl + HNO_3 + H_2O$, Murakami's reagent and Murakami's reagent with Caro's reagent ($H_2SO_4 + H_2O_2$). It was reported that $HCl + HNO_3 + H_2O$ was the higher reduction of Co content while Murakami's reagent with Caro's reagent was the higher of surface roughness. The result for Murakami with Caro' acid was agreeing by R.Polini *et al*. (2000) and Kamiya *et al*. (2001). For reduction of cobalt content, both of them were obtained the same result that the nitric acid was better than Murakami's reagent. Sahoo and Chattopadhyay (2002) study the effect of HNO_3+H_2O, $HNO_3+HCl+H_2O$, and HNO_3+3HCl to cobalt content and surface roughness. It was reported that $HNO_3+HCl+H_2O$ was the best pretreatment in term of reduction of cobalt content, roughening the surface, and improve diamond coating performance. Bu (2009) was found that Caro's reagent better than nitric acid in term of cobalt removal and surface roughness. Tang *et al.* (2002) and Ilias *et al*. (2000) were carried out the high temperature of nitric acid to evaluated better diamond coating.

From the literatures, it can be seen that attempts to study pretreatment at high temperature was still scarce. Hence there is a need to study the high temperature acid which can be expected to reduce the pretreatment time. The aim of this research is to study the effect of pretreatment parameters including time and temperature of acid solution on tungsten carbide in term of cobalt removal and surface roughness substrate.

1.2 Statement of Problem

Pretreatment is important stages affecting to performance of diamond coating. There are quite extensive research have been done by previous researchers on the pretreatment process and one of method have been used was chemical pretreatment. However, most of these studies were time consuming to roughen and remove the cobalt especially on tungsten carbide. Typically, pretreatment process prior to diamond coating was conducted at room temperature. The problem faced in room temperature is this result in longer time for removing cobalt layer using certain etchants. This will be increasing the cost to manufacture the cutting tool which is used in machining process. Increasing the temperature during pretreatment may help to reduce pretreatment time.

1.3 Objective of the Study

The objectives of this study are:

i. To evaluate the effect of pretreatment parameters (temperature and time) on roughness and cobalt removal of WC substrate.

ii. To develop an empirical model to represent relationship between chemical treatment parameters and surface roughness and Co removal.

1.4 Significance of the Study

High temperature in chemical pretreatment process prior diamond coating has generated interest in understanding the behavior of acid etching. Types of acid etching during pre-treatment on tungsten carbide are well documented. However, very little has been reported on effect of temperature during pretreatment prior CVD coating. It is hoped that comprehensive study on this research will enrich knowledge and understanding of pre-treatment procedure. This study also expects that increasing in the temperature of pretreatment could shorter a pretreatment time which is potential to save considerable cost especially for producing coated cutting tool.

1.5 Scopes of the Study

The scopes of the study were:

I. Only 6 % Co tungsten carbide was used as the substrate material.

II. Only the temperature and time of etching solution were varied and while the type of etching solution was fixed to Caro's acid.

III. Design Expert 6 software was used to plan and analyze the experiment.

1.6 Summary

The formation of graphitization on tungsten carbide tool was influenced on quality of diamond coating. It occurs due the present of cobalt content which is used as a binder between tungsten and carbide. There are a lot of methods study were conducted and one of these methods is using acid etching which is not only to remove the cobalt but also to roughen the substrate surface. Surface roughness also influences the quality of diamond coating especially in terms of adhesion strength. There are two types of acid etching that commonly used which are single step and two step etching. Both of etching type's affects adhesion of diamond coating. However a longer etching time is needed for this etching especially in the two steps etching which leads to increasing the cost of manufacture. In this study, the temperature of acid solution was varied to investigate the effect of temperature on cobalt content and surface roughness.